

# INTERNATIONAL SEARCH REPORT

Intern. Application No.  
**PCT/EP 99/10131**

**A. CLASSIFICATION OF SUBJECT MATTER**  
IPC 7 H01B7/295 C09K21/02

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**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)  
IPC 7 H01B C09K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 83 04041 A (NORSK KABELFABRIK AS) 24 November 1983 (1983-11-24)  page 3, line 7-17 page 12, line 5-9; <u>claims 1-34</u>	1-3, 5-15, 18-24
X	EP 0 718 388 A (BALLE ERIKA ;OTTO EUGEN (DE); OTTO UTA DR (DE)) 26 June 1996 (1996-06-26) page 5, line 49-59 page 11, line 49-51; <u>claims 1,13</u>	1-3,6,7, 12,14,18

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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# INTERNATIONAL SEARCH REPORT

information on patent family members

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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<b>(51) International Patent Classification 7 :</b> <b>H01B 7/295, C09K 21/02</b>	<b>A1</b>	<b>(11) International Publication Number:</b> <b>WO 00/39810</b> <b>(43) International Publication Date:</b> <b>6 July 2000 (06.07.00)</b>
<b>(21) International Application Number:</b> PCT/EP99/10131 <b>(22) International Filing Date:</b> 20 December 1999 (20.12.99)  <b>(30) Priority Data:</b> 98124648.1 24 December 1998 (24.12.98) EP 60/115,593 1 December 1999 (01.12.99) US  <b>(71) Applicant (for all designated States except US):</b> PIRELLI CAVI E SISTEMI S.P.A. [IT/IT]; Viale Sarca, 222, I-20126 Milano (IT).  <b>(72) Inventors; and</b> <b>(75) Inventors/Applicants (for US only):</b> PERUZZOTTI, Franco [IT/IT]; Via Liguria, 18, I-20025 Legnano (IT). TIRELLI, Diego [IT/IT]; Via Marsala, 69, I-20099 Sesto San Giovanni (IT). LIBOI, Paolo [IT/IT]; Via Angélo Moro, 3/B, I-20097 San Donato Milanese (IT). ALBIZZATI, Enrico [IT/IT]; Via Trieste, 2, I-28040 Lesa (IT).  <b>(74) Agent:</b> GIANNESI, Pier, Giovanni; Pirelli S.p.A., Industrial Property Dept., Viale Sarca, 222, I-20126 Milan (IT).		<b>(81) Designated States:</b> AE, AL, AU, BA, BB, BG, BR, CA, CN, CU, CZ, EE, GD, GE, HR, HU, ID, IL, IN, IS, JP, KP, KR, LC, LK, LR, LT, LV, MG, MK, MN, MX, NO, NZ, PL, RO, RU, SG, SI, SK, TR, TT, UA, US, UZ, VN, YU, ZA, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> PROCESS FOR PRODUCING SELF-EXTINGUISHING CABLES WITH LOW-LEVEL PRODUCTION OF FUMES, AND FLAME-RETARDANT COMPOSITIONS USED THEREIN		
<b>(57) Abstract</b>  Process for producing cables, in particular electrical cables for low-voltage power transmission or for telecommunications, which have self-extinguishing properties and produce a low level of fumes, in which the flame-retardant coating layer is obtained by extruding a flame-retardant composition comprising a polymer base, an inorganic flame-retardant filler and a dehydrating agent. The presence of the dehydrating agent makes it possible to obtain a smooth and uniform flame-retardant layer which is virtually free of pores.		

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**"PROCESS FOR PRODUCING SELF-EXTINGUISHING CABLES WITH  
LOW-LEVEL PRODUCTION OF FUMES, AND FLAME-RETARDANT  
COMPOSITIONS USED THEREIN"**

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The present invention relates to a process for producing cables, in particular electrical cables for low-voltage power transmission or for telecommunications, these cables having self-extinguishing properties and producing a low level of fumes, and to the flame-retardant compositions used therein.

Self-extinguishing cables are generally produced by extruding over the core of the cable a flame-retardant coating consisting of a polymer composition which has been given flame-resistant properties by the addition of a suitable additive. Polyolefin-based compositions based, for example, on polyethylene or ethylene/vinyl acetate copolymers, containing an organic halide combined with antimony trioxide as flame-retardant additive can, for example, be used for this purpose. However, halogenated flame-retardant additives have many drawbacks since they partially decompose during processing of the polymer, giving rise to halogenated gases that are toxic to workers and corrode the metal parts of the polymer-processing equipment. In addition, when they are placed directly in a flame, their combustion gives rise to very large amounts of fumes containing toxic gases. Similar drawbacks are encountered when polyvinyl chloride (PVC) with added antimony trioxide is used as base polymer.

Thus, in recent years, use has been made of halogen-free compounds in the production of self-extinguishing cables, in which a polymer base, generally of polyolefin type, is mixed with inorganic flame-retardant fillers, generally hydroxides, hydrated oxides or hydrated salts of metals, in particular of aluminium

or magnesium, such as magnesium hydroxide or alumina trihydrate, or mixtures thereof (see, for example, patents US 4,145,404, US 4,673,620, EP 328,051 and EP 530,940).

5           The inorganic fillers can be used as they are or coated with various hydrophobic products, for example with saturated or unsaturated fatty acids or salts thereof, in particular oleic acid or stearic acid or the corresponding oleates or stearates, or with organic  
10   silanes or titanates.

          For example, patent application WO 96/27885 describes a flame-retardant composition for coating electrical cables, comprising polypropylene as polymer matrix supplemented with 1-20 % by weight of a  
15   polyethylene wax and 100-200 % by weight of magnesium hydroxide coated with a hydrophobic product, for example an alkylsilane (% by weight relative to the weight of the polypropylene). This coating is said to increase the compatibility between the filler and the polymer matrix  
20   and at the same time to impart hydrophobic properties to the flame-retardant coating, thus avoiding the absorption of moisture which would reduce the efficiency of the insulating properties of the material.

          Japanese patent application JP-07-161,230  
25   (Kokai) describes polymer compositions with flame-retardant properties, containing appropriately ground natural magnesium hydroxide which has been surface-treated with a fatty acid or a salt thereof, or with a silane or a titanate, in amounts of between 0.5 and 5 %  
30   by weight relative to the weight of the hydroxide. As described in that patent application, surface treatment of the filler is said to make it possible to reduce the absorption of moisture, thus preventing the water vapour released from the filler during the extrusion of the  
35   composition on the cable from effecting a kind of expansion of the material and a worsening of the surface appearance of the cable thus obtained.

The Applicant has observed that, in the production of self-extinguishing cables in which an inorganic filler as described above is used, coating of this filler with hydrophobic agents, as indicated in the prior art, is not sufficient to obtain a satisfactory result which is reproducible on an industrial scale, in particular when the process for extruding the flame-retardant composition is carried out at elevated temperatures in order to increase the fluidity and thus the processibility of the composition so as to obtain high extrusion rates and thus high productivity. Specifically, the Applicant has often observed, with flame-retardant fillers which are either coated or non-coated, and in particular with those of natural origin (i.e. obtained from minerals rather than by synthesis), the formation of a coating layer of unsatisfactory appearance, which has a dull, rough surface. In addition, in certain cases, the formation of pores inside the flame-retardant layer has been observed, with a consequent reduction in the mechanical properties of this coating.

The Applicant has now found that it is possible to obtain a self-extinguishing cable with a flame-retardant coating which is substantially free of pores and which has a smooth and uniform outer surface, if a dehydrating agent is added to the composition comprising a polymer base and an inorganic flame-retardant filler. This dehydrating agent can be added to the flame-retardant composition during the mixing (compounding) phase or directly upstream of the extruder.

In a first aspect, the present invention thus relates to a process for producing self-extinguishing cables with low-level production of fumes, which comprises:

- (a) preparing a flame-retardant composition comprising a polymer base and an inorganic flame-retardant filler;

(b) extruding said flame-retardant composition on an electrical conductor, which is optionally precoated with an insulating layer, so as to obtain a flame-retardant coating layer;

5 characterized in that a dehydrating agent is added to said flame-retardant composition.

According to a first embodiment of the invention, the dehydrating agent is added during phase (a) of preparation of the flame-retardant composition.

10 In a preferred embodiment, the dehydrating agent is added during phase (a) of preparation of the flame-retardant composition after a first phase of mixing the composition at a predetermined temperature and for a predetermined time so as to reduce the moisture content  
15 present in the flame-retardant filler.

According to a further embodiment of the invention, the dehydrating agent is added during phase (b) of extrusion of the flame-retardant composition.

20 According to a further aspect, the present invention relates to a flame-retardant composition comprising a polymer base and an inorganic flame-retardant filler, characterized in that it also comprises a dehydrating agent.

25 In the Applicant's perception, the dehydrating agent exerts its action by absorbing the water present in the flame-retardant filler, which is released during the heating of the composition in the extrusion phase. The mechanism of absorption is preferably of irreversible type, or the dehydrating agent can absorb  
30 the water reversibly but with a low rate of release of the moisture at the extrusion temperature, so as to ensure the virtual absence of water in the vapour state during the extrusion phase. Working in this way prevents the formation of pores inside the flame-retardant  
35 coating and/or the appearance of roughness on its surface. The amount of water released increases as the extrusion temperature increases, as a result of which



the advantages deriving from the presence of the dehydrating agents become particularly evident when relatively high extrusion temperatures, generally above 180°C, preferably above 200°C, are used.

5 In addition, the Applicant has found that the effect of the dehydrating agent on the surface appearance and on the mechanical properties of the flame-retardant coating is particularly evident when flame-retardant fillers of natural origin are used, for  
10 example magnesium hydroxide obtained by grinding minerals such as brucite and the like. The reason for this is thought to be that a flame-retardant filler of natural origin contains large amounts of moisture, greater than the amounts typically found in synthetic  
15 flame-retardant fillers. The moisture present can derive either from the starting mineral or from the grinding process to which this mineral is subjected, or can be absorbed from the surroundings.

Dehydrating agents which can be used are readily  
20 available inorganic compounds which are easy to handle, which do not adversely effect the mechanism of action of the flame-retardant filler and which do not produce toxic products if they are heated to high temperature or exposed to the direct action of a flame. In particular,  
25 the dehydrating agent can be chosen from: calcium oxide, calcium chloride, anhydrous alumina, zeolites, magnesium sulphate, magnesium oxide, barium oxide and the like, or mixtures thereof. Calcium oxide and zeolites, or mixtures thereof, are particularly preferred.

30 The amount of dehydrating agent to be added to the flame-retardant composition is mainly predetermined as a function of the nature and efficacy of this agent and on the amount of water present in the flame-retardant filler. In general, it is believed that an  
35 amount of dehydrating agent of between 0.5 and 15 % by weight, preferably between 1 and 10 % by weight,

relative to the weight of the flame-retardant filler, is sufficient to ensure a satisfactory result.

Flame-retardant fillers which can generally be used are hydroxides, hydrated oxides, salts or hydrated salts of metals, in particular of calcium, aluminium or magnesium, such as: magnesium hydroxide, alumina trihydrate, hydrated magnesium carbonate, magnesium carbonate, hydrated calcium and magnesium carbonate, calcium and magnesium carbonate, or mixtures thereof. Magnesium hydroxide is particularly preferred, since it is characterized by a decomposition temperature of about 340°C and thus allows high extrusion temperatures to be used. It is more particularly preferred to use magnesium hydroxide of natural origin, obtained by grinding minerals based on magnesium hydroxide, such as brucite or the like, as described in European patent application No. 97121042.2, filed on 01.12.97 by the Applicant and in the publication on Research Disclosure No. 407 (March 1998).

The flame-retardant filler is generally used in the form of particles which are untreated or surface-treated with saturated or unsaturated fatty acids containing from 8 to 24 carbon atoms, or metal salts thereof, such as, for example: oleic acid, palmitic acid, stearic acid, isostearic acid, lauric acid; magnesium or zinc stearate or oleate; and the like. In order to increase the compatibility with the polymer matrix, the flame-retardant filler can likewise be surface-treated with suitable coupling agents, for example organic silanes or titanates such as vinyltriethoxysilane, vinyltriacetylsilane, tetraisopropyl titanate, tetra-n-butyl titanate and the like.

The amount of flame-retardant filler to be added is predetermined so as to obtain a cable which is capable of passing the ordinary fire-resistance tests, for example the test according to standards IEC 332-1 and IEC 332.3 A,B,C. In general, this amount is between

10 and 90 % by weight, preferably between 30 and 80 % by weight, relative to the total weight of the flame-retardant composition.

5 The polymer base can generally be chosen from: polyolefins, various olefin copolymers, copolymers of olefins with ethylenically unsaturated esters, polyesters, polyethers, polyether/polyester copolymers, and mixtures thereof.

Examples of such polymers are: high-density  
10 polyethylene (HDPE) ( $d=0.940-0.970 \text{ g/cm}^3$ ), medium-density polyethylene (MDPE) ( $d=0.926-0.940 \text{ g/cm}^3$ ), low-density polyethylene (LDPE) ( $d=0.910-0.926 \text{ g/cm}^3$ );  
15 copolymers of ethylene with  $\alpha$ -olefins containing from 3 to 12 carbon atoms (for example 1-butene, 1-hexene, 1-octene and the like), in particular linear low-density polyethylene (LLDPE) and ultra-low-density polyethylene (ULDPE) ( $d=0.860-0.910 \text{ g/cm}^3$ ); polypropylene (PP);  
20 thermoplastic copolymers of propylene with another olefin, in particular ethylene; copolymers of ethylene with at least one ester chosen from alkyl acrylates, alkyl methacrylates and vinyl carboxylates, in which the linear or branched alkyl group can contain from 1 to 8, preferably from 1 to 4, carbon atoms, while the linear or branched carboxylic group can contain from 2 to 8,  
25 preferably from 2 to 5, carbon atoms, in particular ethylene/vinyl acetate (EVA) copolymers; ethylene/ethyl acrylate (EEA) copolymers, ethylene/butyl acrylate (EBA) copolymers; ethylene/ $\alpha$ -olefin rubbers, in particular ethylene/propylene rubbers (EPR),  
30 ethylene/propylene/diene rubbers (EPDM); natural rubber; butyl rubbers; and mixtures thereof.

Copolymers which are particularly preferred are those which can be obtained by copolymerization of  
35 ethylene with at least one  $\alpha$ -olefin containing from 3 to 12 carbon atoms, and optionally with a diene, in the presence of a "single-site" catalyst, in particular a metallocene catalyst or a constrained geometry catalyst.

These copolymers are characterized by a density of between 0.860 and 0.904 g/cm<sup>3</sup>, preferably from 0.865 to 0.902 g/cm<sup>3</sup>, and by a composition distribution index greater than 45 %, said index being defined as the percentage by weight of the copolymer molecules having an  $\alpha$ -olefin content of up to 50 % of the total average molar content of  $\alpha$ -olefin. These copolymers preferably have the following monomer composition: 75-97 mol%, preferably 90-95 mol%, of ethylene; 3-25 mol%, preferably 5-10 mol%, of  $\alpha$ -olefin; 0-5 mol%, preferably 0-2 mol%, of a diene. The  $\alpha$ -olefin is preferably chosen from propylene, 1-butene, 1-hexene, 1-octene and the like. Products of this type are commercially available under the tradenames Engage® from Du Pont-Dow Elastomers and Exact® from Exxon Chemical.

The ethylene copolymers obtained by single-site catalysis are preferably used as a mixture with a crystalline propylene homopolymer or copolymer, as described, for example, in the abovementioned European patent application No. 97121042.2, or with an ethylene homopolymer or copolymer which has a density of between 0.905 and 0.970 g/cm<sup>3</sup>, preferably between 0.910 and 0.940 g/cm<sup>3</sup>, as described, for example, in European patent application No. 98118194.4 filed on 25.9.98 in the name of the Applicant, or alternatively in US patent 5,707,732. In particular, the polymer base preferably comprises from 5 to 60 % by weight, more preferably from 10 to 45 % by weight, of a propylene or ethylene homopolymer or copolymer as defined above, and from 40 to 95 % by weight, more preferably from 55 to 90 % by weight, of an ethylene copolymer obtained by single-site catalysis, the percentages being relative to the total weight of the polymeric components (a) and (b).

A coupling agent capable of increasing the interaction between the active groups of the flame-retardant filler and the polymer chains may be added to the mixture in order to enhance the compatibility

between the flame-retardant filler and the polymer matrix. This coupling agent can be chosen from those known in the art, for example: saturated silane compounds or silane compounds containing at least one ethylenic unsaturation; epoxides containing an ethylenic unsaturation; monocarboxylic acids or, preferably, dicarboxylic acids having at least one ethylenic unsaturation, or derivatives thereof, in particular anhydrides or esters.

Examples of silane compounds which are suitable for this purpose are:  $\gamma$ -methacryloxypropyltrimethoxysilane, allyltrimethoxysilane, allyltriethoxysilane, allylmethyldimethoxysilane, allylmethyldiethoxysilane, methyltriethoxysilane, methyltris(2-methoxyethoxy)silane, dimethyldiethoxysilane, vinyltris(2-methoxyethoxy)silane, vinyltrimethoxysilane, vinylmethyldimethoxysilane, vinyltriethoxysilane, octyltriethoxysilane, isobutyltriethoxysilane, isobutyltrimethoxysilane and the like, or mixtures thereof.

Examples of epoxides containing an ethylenic unsaturation are: glycidyl acrylate, glycidyl methacrylate, monoglycidyl ester of itaconic acid, glycidyl ester of maleic acid, vinyl glycidyl ether, allyl glycidyl ether and the like, or mixtures thereof.

Monocarboxylic or dicarboxylic acids, having at least one ethylenic unsaturation, or derivatives thereof, which can be used as coupling agents are, for example: maleic acid, maleic anhydride, fumaric acid, citraconic acid, itaconic acid, acrylic acid, methacrylic acid and the like, and anhydrides or esters derived from these, or mixtures thereof. Maleic anhydride is particularly preferred.

The coupling agents can be used as they are or pregrafted onto a polyolefin, for example polyethylene or copolymers of ethylene with an  $\alpha$ -olefin, by means of a radical reaction (see for example patent EP-530,940).

The amount of coupling agent grafted is generally between 0.05 and 5 parts by weight, preferably between 0.1 and 2 parts by weight, relative to 100 parts by weight of polyolefin. Polyolefins grafted with maleic anhydride are available as commercial products known, for example, under the brand names Fusabond® (Du Pont), Orevac® (Elf Atochem), Exxelor® (Exxon Chemical), Yparex® (DSM), etc.

Alternatively, the coupling agents of carboxylic or epoxide type mentioned above (for example maleic anhydride) or the silanes with ethylenic unsaturation (for example vinyltrimethoxysilane) can be added to the mixture in combination with a radical initiator so as to graft the compatibilizing agent directly onto the polymer matrix. An organic peroxide such as tert-butyl perbenzoate, dicumyl peroxide, benzoyl peroxide, di-tert-butyl peroxide and the like can, for example, be used as initiator. This method is described, for example, in patent US-4,317,765, in Japanese patent application JP-62-58774 or alternatively in the abovementioned European patent applications Nos. 97121042.2 and 98118194.4.

The amount of coupling agent to be added to the mixture can vary mainly depending on the type of coupling agent used and on the amount of flame-retardant filler added, and is generally between 0.01 and 5%, preferably between 0.05 and 2%, by weight relative to the total weight of the base polymer mixture.

Other conventional components such as antioxidants, processing coadjuvants, lubricants, pigments, other fillers and the like can be added to the compositions according to the present invention.

Conventional antioxidants which are suitable for this purpose are, for example:

polymerized trimethyldihydroquinoline, 4,4'-thiobis (3-methyl-6-tert-butyl)phenol; pentaerythryl tetra-[3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate], 2,2'-

thiodiethylene bis[3-(3,5-di-tert-butyl-4-hydroxy-phenyl)propionate] and the like, or mixtures thereof.

Other fillers which may be used in the present invention include, for example, glass particles, glass  
5 fibres, calcined kaolin, talc and the like, or mixtures thereof. Processing co-adjuvants usually added to the polymer base are, for example, calcium stearate, zinc stearate, stearic acid, paraffin wax, silicone rubbers and the like, or mixtures thereof.

10 The flame-retardant compositions according to the present invention are preferably used in non-crosslinked form, in order to obtain a coating with thermoplastic properties which is thus recyclable.

The flame-retardant compositions according to  
15 the present invention can be prepared by mixing the polymer base, the flame-retardant filler, the dehydrating agent and the other additives which may be present according to techniques known in the art, for example using an internal mixer of the type containing  
20 tangential rotors (Banbury) or interlocking rotors, or in continuous mixers of the Ko-Kneader (Buss) type or of the co-rotating or counter-rotating twin-screw type.

Preferably, the dehydrating agent is introduced after a first phase of processing the composition during  
25 which, on account of the heating generated by the compounding process, the flame-retardant filler loses a certain amount of the moisture absorbed. In this way, premature depletion of the water-absorbing capacity of the dehydrating filler is avoided, this filler needing  
30 to be active mainly during the subsequent extrusion phase. The temperature of the composition in this first compounding phase is at least 100°C, preferably at least 150°C, and is carried out for a period of at least 5 minutes.

35 Alternatively, rather than adding the dehydrating agent during the phase of preparation of the

flame-retardant composition, it can be added during the extrusion phase, for example via the extruder hopper.

In both cases, the dehydrating agent can be added to the flame-retardant composition in divided form (granules, powder), optionally coated with dispersing and protective agents, such as microwaxes, fatty acids and the like. Alternatively, in order to improve its dispersion in the polymer base, the dehydrating agent can be used predispersed in a polymer material (for example a semi-crystalline ethylene/propylene rubber).

During the extrusion phase, the flame-retardant compositions thus obtained can be used to coat the conductor directly, or to make an outer sheath on the conductor which has been precoated with an insulating layer. When two layers are present, the extrusion can take place in two separate phases, the inner layer being extruded on the conductor in a first passage and the outer layer being extruded on the inner layer in a second passage. Advantageously, the coating process can take place in a single passage, for example by means of the "tandem" technique, in which two separate extruders arranged in series are used, or alternatively by co-extrusion with a single extrusion head.

The temperature at which the flame-retardant composition is extruded can vary within a wide range and is predetermined as a function of the extrusion rate to be obtained. The extrusion rate in fact depends on the viscosity of the composition in the molten state and thus on its temperature. In turn, the viscosity depends mainly on the type of polymer base and on the type and amount of flame-retardant filler used. The minimum extrusion temperature for the composition is generally not less than the plasticization temperature of the polymer base, while the maximum extrusion temperature is predetermined so as to avoid degradation or decomposition of the polymer base and/or of the flame-retardant filler. Thus, on the basis of the



abovementioned criteria, in the case of flame-retardant compositions based on a mixture of polypropylene and ethylene/ $\alpha$ -olefin copolymers as described above, in which magnesium hydroxide is used as flame-retardant filler, the temperature at which the flame-retardant composition is extruded is generally between 160°C and 320°C, preferably between 200°C and 280°C.

Although the present description is mainly directed towards the production of self-extinguishing cables by extrusion, the advantages deriving from the use of the dehydrating agent according to the present invention can be evident in different extrusion or moulding processes for the general production of rubber articles in which hygroscopic fillers are used, for example junction boxes for electrical cable junctions or terminals, in particular when high processing temperatures are required in order to obtain increased fluidity of the material to be extruded or moulded.

Some examples of embodiments will now be reported for the purpose of illustrating the present invention more clearly, with particular reference to the attached:

Figure 1, which is a schematic drawing in cross-section of a self-extinguishing, low-voltage unipolar electrical cable which can be made according to the present invention.

The term "low-voltage" generally means a voltage of less than 2 kV, preferably less than 1 kV.

The cable in Fig. 1 comprises a conductor (1), an inner layer (2) which functions as an electrical insulator, and an outer layer (3) which functions as a protective sheath with flame-retardant properties.

The inner layer (2) can consist of a crosslinked or non-crosslinked, halogen-free polymer composition with electrical insulation properties, which is known in the art, chosen, for example, from: polyolefins (homopolymers or copolymers of various olefins),

olefin/ethylenically unsaturated ester copolymers, polyesters, polyethers, polyether/polyester copolymers and mixtures thereof. Examples of such polymers are: polyethylene (PE), in particular linear low-density PE (LLDPE); polypropylene (PP); propylene/ethylene thermoplastic copolymers; ethylene/propylene rubbers (EPR) or ethylene/propylene/diene rubbers (EPDM); natural rubbers; butyl rubbers; ethylene/vinyl acetate (EVA) copolymers; ethylene/methyl acrylate (EMA) copolymers; ethylene/ethyl acrylate (EEA) copolymers; ethylene/butyl acrylate (EBA) copolymers; ethylene/ $\alpha$ -olefin copolymers, and the like.

Alternatively, a self-extinguishing cable which can be made according to the present invention can consist of a conductor coated directly with the flame-retardant composition, without interposition of other insulating layers. In this way, the flame-retardant coating also functions as an electrical insulator. A thin polymer layer which functions as an anti-abrasive agent, optionally combined with a suitable pigment in order to give a coloration for identification purposes, can then be added externally.

#### Preparation of the flame-retardant compositions

The flame-retardant compositions were prepared in a closed Banbury mixer (mixing chamber volume: 1200 cm<sup>3</sup>) filled to a volumetric level of 95 %. The mixing was carried out in two phases. In the first phase, the components of the compound, with the exception of the dehydrating agent, were mixed together until a temperature of about 200°C was reached, so as to ensure good dispersion of the components and to reduce the amount of moisture present in the filler. The dehydrating agent was then added, while keeping the mixing temperature at about 200°C.

#### Mechanical properties

Self-extinguishing cables were produced by extruding the compositions prepared as described above

on a wire of red copper (cross-section  $2.5 \text{ mm}^2$ ) in an extruder with a cylinder 120 mm in diameter and with a length equal to 25 diameters (final thickness of the flame-retardant layer: 0.8 mm). The temperature of the composition in the extruder was kept at about  $250^\circ\text{C}$ , with an extrusion rate of 900 m/min.

The flame-retardant coatings thus obtained were subjected to mechanical tensile strength tests according to CEI standard 20-34 § 5.1. The results are given in Table 1, as the average value obtained over five samples taken at random from each cable produced. All of the cables produced passed the flame-resistance test according to IEC standard 332-1, which consists in subjecting a 60 cm long sample, placed vertically, to the direct action of a Bunsen-burner flame applied for 1 min at an angle of  $45^\circ$  relative to the sample.

TABLE 1

Example	1	2	3	4 (*)	5	6 (*)
Engage® 8003	85	85	85	85	85	85
Moplen® EP1X35HF	15	15	15	15	15	15
Hydrofy® G 1.5	210	210	210	210	--	--
Hydrofy® G 1.5S	--	--	--	--	210	210
Peroximon® DC40	0.4	0.4	0.4	0.4	0.4	0.4
Silquest® A-172	1.8	1.8	1.8	1.8	1.8	1.8
Irganox® 1010	0.8	0.8	0.8	0.8	0.8	0.8
Irganox® MD1024	0.3	0.3	0.3	0.3	0.3	0.3
Stearic acid	1.5	1.5	1.5	1.5	1.5	1.5
Kezadol® GR	2	6	10	--	6	--
Mechanical properties on cable extruded at 900 m/min						
Breaking load (MPa)	14.0	13.8	12.2	14.2	13.5	13.0
Elongation at break (%)	122	140	159	90	157	105

5 (\*) comparative

Engage® 8003 - ethylene/1-octene copolymer obtained by  
metallocene catalysis:

ethylene/1-octene weight ratio = 82/18 (5.5 mol% of 1-octene);  $d = 0.885 \text{ g/cm}^3$ ; MFI = 1.0 g/10'; CDI > 70 %;  $\Delta H_{2f} = 55.6 \text{ J/g}$ ;

5 Moplen® EP1X35HF - random crystalline propylene/ethylene copolymer:

$d = 0.900 \text{ g/cm}^3$ ; MFI = 9.0 g/10';  $T_{2f} = 154^\circ\text{C}$ ;  $\Delta H_{2f} = 90.6 \text{ J/g}$ ;

10 Hydrofy® G 1.5 - natural magnesium hydroxide, obtained by grinding brucite, not surface-treated (SIMA company) with specific surface area:  $10.4 \text{ m}^2/\text{g}$ ;

Hydrofy® G 1.5S - natural magnesium hydroxide, obtained by grinding brucite, surface-treated with stearic acid (SIMA company) with specific surface area:  $10.4 \text{ m}^2/\text{g}$ ;

15 Silquest® A-172 - coupling agent:

vinyltris(2-methoxyethoxy)silane (VTMOEO);

Peroximon® DC40 - peroxidic initiator: dicumyl peroxide;

Irganox® 1010 - antioxidant:

20 pentaerythryl tetrakis[3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate] (Ciba-Geigy);

Irganox® MD1024 - metal deactivator: 1,2-bis(3,5-di-tert-butyl-4-hydroxyhydrocinnamoyl)hydrazine (Ciba-Geigy);

25 Kezadol® GR - calcium oxide predispersed in semicrystalline EPR rubber (80 % by weight of CaO), in the form of granules with average diameter 6-7  $\mu\text{m}$  (Kettlitz company).

30 The results given in Table 1 demonstrate clearly that in the cables produced in the presence of calcium oxide, the flame-retardant coating has excellent mechanical properties. In contrast, the comparative cables, produced without dehydrating agent, show unsatisfactory mechanical properties and are not capable of passing the specifications required for cables of this type (usually a breaking load greater than 12.5 MPa and an elongation at break greater than 125 %). Under visual examination, the comparative cables show a dull

surface with the presence of micropores inside the flame-retardant coating, these defects being entirely absent from the cables produced according to the invention.

CLAIMS

1. Process for producing self-extinguishing cables with low-level production of fumes, which comprises:
  - 5 (a) preparing a flame-retardant composition comprising a polymer base and an inorganic flame-retardant filler;
  - (b) extruding said flame-retardant composition on an electrical conductor, which is optionally precoated with an insulating layer, so as to obtain a flame-retardant coating;
  - 10 characterized in that a dehydrating agent is added to said flame-retardant composition.
- 15 2. Process according to Claim 1, in which the dehydrating agent is added during phase (a) of preparation of the flame-retardant composition.
- 20 3. Process according to Claim 2, in which the dehydrating agent is added during phase (a) of preparation of the flame-retardant composition after a first phase of mixing the composition at a predetermined temperature and for a predetermined time so as to reduce the moisture content present in the flame-retardant  
25 filler.
4. Process according to Claim 1, in which the dehydrating agent is added during phase (b) of extrusion of the flame-retardant composition.  
30
5. Process according to any one of the preceding claims, in which the dehydrating agent is added to the flame-retardant composition in divided form.
- 35 6. Process according to any one of the preceding claims, in which the dehydrating agent is added to the

flame-retardant composition after first being dispersed in a polymer material.

- 5 7. Process according to any one of the preceding claims, in which the flame-retardant composition is extruded at a predetermined temperature which depends on the extrusion rate to be obtained.
- 10 8. Process according to Claim 7, in which the flame-retardant composition is extruded at a temperature of between 160°C and 320°C.
- 15 9. Process according to Claim 8, in which the flame-retardant composition is extruded at a temperature of between 200°C and 280°C.
- 20 10. Process according to any one of the preceding claims, in which the dehydrating agent is chosen from: calcium oxide, calcium chloride, anhydrous alumina, zeolites, magnesium sulphate, magnesium oxide, barium oxide, and the like, or mixtures thereof.
- 25 11. Process according to Claim 10, in which the dehydrating agent is chosen from: calcium oxide and zeolites, or mixtures thereof.
- 30 12. Process according to any one of the preceding claims, in which the dehydrating agent is added to the flame-retardant composition in an amount of between 0.5 and 15 % by weight relative to the weight of the flame-retardant filler.
- 35 13. Process according to Claim 12, in which the dehydrating agent is added to the flame-retardant composition in an amount of between 1 and 10 % by weight relative to the weight of the flame-retardant filler.



14. Process according to any one of the preceding claims, in which the flame-retardant filler is chosen from: hydroxides, hydrated oxides, salts or hydrated salts of metals.

5

15. Process according to Claim 14, in which the flame-retardant filler is chosen from: magnesium hydroxide, alumina trihydrate, hydrated magnesium carbonate, magnesium carbonate, hydrated calcium and magnesium carbonate, calcium and magnesium carbonate, or mixtures thereof.

10

16. Process according to Claim 15, in which the flame-retardant filler is magnesium hydroxide.

15

17. Process according to Claim 15, in which the flame-retardant filler is magnesium hydroxide of natural origin.

20

18. Flame-retardant composition comprising a polymer base and an inorganic flame-retardant filler, characterized in that it also comprises a dehydrating agent.

25

19. Composition according to Claim 18, in which the dehydrating agent is chosen from: calcium oxide, calcium chloride, anhydrous alumina, zeolites, magnesium sulphate, magnesium oxide, barium oxide, and the like, or mixtures thereof.

30

20. Composition according to Claim 19, in which the dehydrating agent is chosen from: calcium oxide and zeolites, or mixtures thereof.

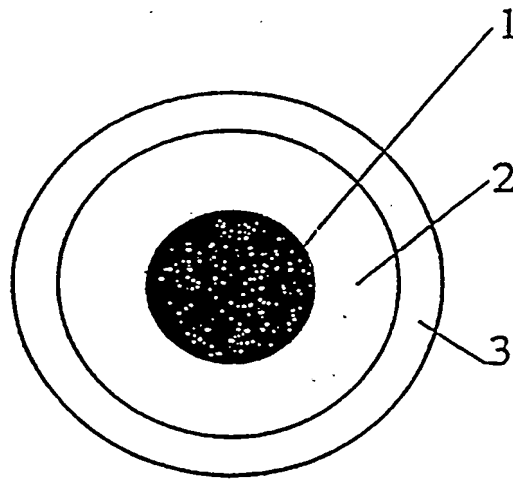
35

21. Composition according to any one of Claims 18 to 20, in which the dehydrating agent is present in an

amount of between 0.5 and 15 % by weight relative to the weight of the flame-retardant filler.

22. Composition according to Claim 21, in which the  
5 dehydrating agent is present in an amount of between 1 and 10 % by weight relative to the weight of the flame-retardant filler.
23. Composition according to any one of Claims 18 to  
10 22, in which the flame-retardant filler is chosen from: hydroxides, hydrated oxides, salts or hydrated salts of metals.
24. Composition according to Claim 23, in which the  
15 flame-retardant filler is chosen from: magnesium hydroxide, alumina trihydrate, hydrated magnesium carbonate, magnesium carbonate, hydrated calcium and magnesium carbonate, calcium and magnesium carbonate, or mixtures thereof.
- 20 25. Composition according to Claim 24, in which the flame-retardant filler is magnesium hydroxide.
26. Composition according to Claim 25, in which the  
25 flame-retardant filler is magnesium hydroxide of natural origin.

FIG. 1



## INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 99/10131

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 H01B7/295 C09K21/02

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 H01B C09K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 83 04041 A (NORSK KABELFABRIK AS) 24 November 1983 (1983-11-24)  page 3, line 7-17 page 12, line 5-9; claims 1-34	1-3, 5-15, 18-24
X	EP 0 718 388 A (BALLE ERIKA ; OTTO EUGEN (DE); OTTO UTA DR (DE)) 26 June 1996 (1996-06-26) page 5, line 49-59 page 11, line 49-51; claims 1,13	1-3,6,7, 12,14,18

☐ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

## \* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"Z" document member of the same patent family

Date of the actual completion of the international search

6 Apr11 2000

Date of mailing of the international search report

13/04/2000

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# INTERNATIONAL SEARCH REPORT

information on patent family members

International Application No

PCT/EP 99/10131

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 8304041 A	24-11-1983	AT 29149 T	15-09-1987
		AU 552976 B	26-06-1986
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		DE 4444160 A	13-06-1996
		JP 8165468 A	25-06-1996

09/868 997

## PATENT COOPERATION TREATY

PCT

REC'D 29 JAN 2001

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PCT

## INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

15

Applicant's or agent's file reference PC792	<b>FOR FURTHER ACTION</b> See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/EP99/10131	International filing date (day/month/year) 20/12/1999	Priority date (day/month/year) 24/12/1998
International Patent Classification (IPC) or national classification and IPC H01B7/295		
Applicant PIRELLI CAVI E SISTEMI S.p.a et al		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.



2. This REPORT consists of a total of 6 sheets, including this cover sheet.

- ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 14 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☒ Certain defects in the international application
- VIII ☒ Certain observations on the international application

Date of submission of the demand  18/07/2000	Date of completion of this report  25.01.2001
Name and mailing address of the international preliminary examining authority:   European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer  Lohberger, S  Telephone No. +49 89 2399 6723 

# INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/EP99/10131

## I. Basis of the report

1. This report has been drawn on the basis of *(substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments (Rules 70.16 and 70.17).):*

### Description, pages:

1-4,13,15-18	as originally filed			
5-12,14	as received on	14/12/2000	with letter of	12/12/2000

### Claims, No.:

1-32	as received on	14/12/2000	with letter of	12/12/2000
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### Drawings, sheets:

1/1	as originally filed
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2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. PCT/EP99/10131

- ☐ the description, pages:  
☐ the claims, Nos.:  
☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

*(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)*

6. Additional observations, if necessary:

**V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

**1. Statement**

Novelty (N)	Yes:	Claims	1-32
	No:	Claims	
Inventive step (IS)	Yes:	Claims	1-32
	No:	Claims	
Industrial applicability (IA)	Yes:	Claims	1-32
	No:	Claims	

- 2. Citations and explanations**  
**see separate sheet**

**VII. Certain defects in the international application**

The following defects in the form or contents of the international application have been noted:  
**see separate sheet**

**VIII. Certain observations on the international application**

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:  
**see separate sheet**



**Re Item V**

**Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

1. Reference is made to the following document/s/:  
D1: WO 83 04041 A (NORSK KABELFABRIK AS) 24 November 1983 (1983-11-24)  
D2: EP-A-0 718 388 (BALLE ERIKA ;OTTO EUGEN (DE); OTTO UTA DR (DE))  
26 June 1996 (1996-06-26)
2. D1, especially description page 3, lines 7 to 17, page 8, line 19 to page 9, line 4, examples and claims discloses a process and a composition for producing a self-extinguishing cable similar to claims 1 or 30.  
D1 uses in the composition an organic polymer base and an inorganic filler. A dehydrating agent (CaO, "drying agent") is added. The composition is extruded around a conductor to form a self-extinguishing cable with low-level production of fumes. The purpose for the addition of the "drying agent" is the same as in present Application, namely to avoid the formation of pores during extrusion and to get a more homogeneous mass.  
D1 however is silent about any preheating step of polymer base and inorganic filler in order to remove moisture. It was not evident for a skilled man to preheat the filler/polymer mixture in the light of the teachings of D1. This preheating leads to the advantage of no premature depletion of the water absorbing capacity of the dehydrating agent.  
Applicant's argumentation with regard to D1 is therefore convincing. Claims 1 and 30 are in accordance with article 33(2) and (3) PCT over D1.  
However see section VII.
3. Claims 2 to 17 are preferred embodiments of claim 1 and as such they fulfil the requirements of article 33 (2) and (3) PCT as well.

Claim 31 is a preferred embodiment of claim 30 and as such it fulfils the requirements of article 33 (2) and (3) PCT as well.

4. D2, especially description page 5, lines 49 to 59, page 11, lines 49 to 51, examples and claims discloses a flame-retardant composition for a cable as set out in present claim 18 (polymer base, inorganic flame-retardant filler and dehydrating agent). D2 does neither mention the specific polymer base of claim 18 nor the specific filler. Additionally D2 is silent about any preheating step of a filler-polymer mixture. Therefore D2 no longer is considered to be of relevance for present set of claims.
5. The composition of claim 18 is not mentioned in D1 either. Since the process of D1 does not teach the preheating is cannot be considered to be evident for a skilled man to replace the low-moisture-containing fillers of D1 by the natural magnesium hydroxide of claim 18.  
Therefore claim 18 is acceptable under article 33(2) and (3) over D1 as well.
6. Claims 19 to 29 are preferred embodiments of claim 18 and as such they fulfil the requirements of article 33 (2) and (3) PCT as well.
7. Claim 32 which relates to a cable which is coated by the composition of claim 18 is acceptable for the same reasons as already set out for claim 18.

### **Re Item VII**

#### **Certain defects in the international application**

1. The amendments filed with the letter dated 12.12.00 with regard to claims 1 and 30 introduce subject-matter which extends beyond the content of the application as filed, contrary to Article 34(2)(b) PCT. The amendments concerned are the following: the feature of a "predetermined temperature for a predetermined time" has no basis in the Application as filed. Page 11, lines 23 to 31 only covers the feature a first phase of heating so that the filler loses a certain amount of moisture.

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT - SEPARATE SHEET**

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International application No. PCT/EP99/10131

2. Page 11, line 23 of present description is adapted to claim 1. The word "preferably" should have been cancelled.

**Re Item VIII**

**Certain observations on the international application**

1. The scope of claims 1 and 30 is not clear. The expression "mixing ...at a predetermined temperature for a predetermined time so as to reduce moisture" is vague and undefined. Besides the article 34(2)b objection claims 1 and 30 must as well be objected under article 6 PCT.

the advantages deriving from the presence of the dehydrating agents become particularly evident when relatively high extrusion temperatures, generally above 180°C, preferably above 200°C, are used.

5           In addition, the Applicant has found that the effect of the dehydrating agent on the surface appearance and on the mechanical properties of the flame-retardant coating is particularly evident when flame-retardant fillers of natural origin are used, for  
10   example magnesium hydroxide obtained by grinding minerals such as brucite. The reason for this is thought to be that a flame-retardant filler of natural origin contains large amounts of moisture, greater than the amounts typically found in synthetic flame-retardant  
15   fillers. The moisture present can derive either from the starting mineral or from the grinding process to which this mineral is subjected, or can be absorbed from the surroundings.

          Dehydrating agents which can be used are readily  
20   available inorganic compounds which are easy to handle, which do not adversely effect the mechanism of action of the flame-retardant filler and which do not produce toxic products if they are heated to high temperature or exposed to the direct action of a flame. In particular,  
25   the dehydrating agent can be chosen from: calcium oxide, calcium chloride, anhydrous alumina, zeolites, magnesium sulphate, magnesium oxide, barium oxide, or mixtures thereof. Calcium oxide and zeolites, or mixtures thereof, are particularly preferred.

30           The amount of dehydrating agent to be added to the flame-retardant composition is mainly predetermined as a function of the nature and efficacy of this agent and on the amount of water present in the flame-retardant filler. In general, it is believed that an  
35   amount of dehydrating agent of between 0.5 and 15 % by weight, preferably between 1 and 10 % by weight,

relative to the weight of the flame-retardant filler, is sufficient to ensure a satisfactory result.

Flame-retardant fillers which can generally be used are hydroxides, hydrated oxides, salts or hydrated salts of metals, in particular of calcium, aluminium or magnesium, such as: magnesium hydroxide, alumina trihydrate, hydrated magnesium carbonate, magnesium carbonate, hydrated calcium and magnesium carbonate, calcium and magnesium carbonate, or mixtures thereof. Magnesium hydroxide is particularly preferred, since it is characterized by a decomposition temperature of about 340°C and thus allows high extrusion temperatures to be used. It is more particularly preferred to use magnesium hydroxide of natural origin, obtained by grinding minerals based on magnesium hydroxide, such as brucite, as described in European patent application WO 99/05688, filed on 01.12.97 by the Applicant and in the publication on Research Disclosure No. 407 (March 1998).

The flame-retardant filler is generally used in the form of particles which are untreated or surface-treated with saturated or unsaturated fatty acids containing from 8 to 24 carbon atoms, or metal salts thereof, such as, for example: oleic acid, palmitic acid, stearic acid, isostearic acid, lauric acid; magnesium or zinc stearate or oleate. In order to increase the compatibility with the polymer matrix, the flame-retardant filler can likewise be surface-treated with suitable coupling agents, for example organic silanes or titanates such as vinyltriethoxysilane, vinyltriacetylsilane, tetraiso-propyl titanate, tetra-n-butyl titanate.

The amount of flame-retardant filler to be added is predetermined so as to obtain a cable which is capable of passing the ordinary fire-resistance tests, for example the test according to standards IEC 332-1 and IEC 332.3 A,B,C. In general, this amount is between 10 and 90 % by weight, preferably between 30 and 80 % by

weight, relative to the total weight of the flame-retardant composition.

The polymer base can generally be chosen from: polyolefins, various olefin copolymers, copolymers of  
5 olefins with ethylenically unsaturated esters, polyesters, polyethers, polyether/polyester copolymers, and mixtures thereof.

Examples of such polymers are: high-density polyethylene (HDPE) ( $d=0.940-0.970$  g/cm<sup>3</sup>), medium-  
10 density polyethylene (MDPE) ( $d=0.926-0.940$  g/cm<sup>3</sup>), low-density polyethylene (LDPE) ( $d=0.910-0.926$  g/cm<sup>3</sup>); copolymers of ethylene with  $\alpha$ -olefins containing from 3 to 12 carbon atoms (for example 1-butene, 1-hexene, 1-octene), in particular linear low-density polyethylene  
15 (LLDPE) and ultra-low-density polyethylene (ULDPE) ( $d=0.860-0.910$  g/cm<sup>3</sup>); polypropylene (PP); thermoplastic copolymers of propylene with another olefin, in particular ethylene; copolymers of ethylene with at least one ester chosen from alkyl acrylates, alkyl  
20 methacrylates and vinyl carboxylates, in which the linear or branched alkyl group can contain from 1 to 8, preferably from 1 to 4, carbon atoms, while the linear or branched carboxylic group can contain from 2 to 8, preferably from 2 to 5, carbon atoms, in particular  
25 ethylene/vinyl acetate (EVA) copolymers; ethylene/ethyl acrylate (EEA) copolymers, ethylene/butyl acrylate (EBA) copolymers; ethylene/ $\alpha$ -olefin rubbers, in particular ethylene/propylene  
30 butyl rubbers; and mixtures thereof.

Copolymers which are particularly preferred are those which can be obtained by copolymerization of ethylene with at least one  $\alpha$ -olefin containing from 3 to 12 carbon atoms, and optionally with a diene, in the  
35 presence of a "single-site" catalyst, in particular a metallocene catalyst or a constrained geometry catalyst. These copolymers are characterized by a density of

between 0.860 and 0.904 g/cm<sup>3</sup>, preferably from 0.865 to 0.902 g/cm<sup>3</sup>, and by a composition distribution index greater than 45 %, said index being defined as the percentage by weight of the copolymer molecules having an  $\alpha$ -olefin content of up to 50 % of the total average molar content of  $\alpha$ -olefin. These copolymers preferably have the following monomer composition: 75-97 mol%, preferably 90-95 mol%, of ethylene; 3-25 mol%, preferably 5-10 mol%, of  $\alpha$ -olefin; 0-5 mol%, preferably 0-2 mol%, of a diene. The  $\alpha$ -olefin is preferably chosen from propylene, 1-butene, 1-hexene, 1-octene. Products of this type are commercially available under the tradenames Engage® from Du Pont-Dow Elastomers and Exact® from Exxon Chemical.

The ethylene copolymers obtained by single-site catalysis are preferably used as a mixture with a crystalline propylene homopolymer or copolymer, as described, for example, in the abovementioned European patent application No. 97121042.2, or with an ethylene homopolymer or copolymer which has a density of between 0.905 and 0.970 g/cm<sup>3</sup>, preferably between 0.910 and 0.940 g/cm<sup>3</sup>, as described, for example, in European patent application No. 98118194.4 filed on 25.9.98 in the name of the Applicant, or alternatively in US patent 5,707,732. In particular, the polymer base preferably comprises from 5 to 60 % by weight, more preferably from 10 to 45 % by weight, of a propylene or ethylene homopolymer or copolymer as defined above, and from 40 to 95 % by weight, more preferably from 55 to 90 % by weight, of an ethylene copolymer obtained by single-site catalysis, the percentages being relative to the total weight of the polymeric components (a) and (b).

A coupling agent capable of increasing the interaction between the active groups of the flame-retardant filler and the polymer chains may be added to the mixture in order to enhance the compatibility between the flame-retardant filler and the polymer

matrix. This coupling agent can be chosen from those known in the art, for example: saturated silane compounds or silane compounds containing at least one ethylenic unsaturation; epoxides containing an ethylenic unsaturation; monocarboxylic acids or, preferably, dicarboxylic acids having at least one ethylenic unsaturation, or derivatives thereof, in particular anhydrides or esters.

Examples of silane compounds which are suitable for this purpose are:  $\gamma$ -methacryloxypropyltrimethoxysilane, allyltrimethoxysilane, allyltriethoxysilane, allylmethyldimethoxysilane, allylmethyldiethoxysilane, methyltriethoxysilane, methyltris-(2-methoxyethoxy)silane, dimethyldiethoxysilane, vinyltris(2-methoxyethoxy)silane, vinyltrimethoxysilane, vinylmethyldimethoxysilane, vinyltriethoxysilane, octyltriethoxysilane, isobutyltriethoxysilane, isobutyltrimethoxysilane, or mixtures thereof.

Examples of epoxides containing an ethylenic unsaturation are: glycidyl acrylate, glycidyl methacrylate, monoglycidyl ester of itaconic acid, glycidyl ester of maleic acid, vinyl glycidyl ether, allyl glycidyl ether, or mixtures thereof.

Monocarboxylic or dicarboxylic acids, having at least one ethylenic unsaturation, or derivatives thereof, which can be used as coupling agents are, for example: maleic acid, maleic anhydride, fumaric acid, citraconic acid, itaconic acid, acrylic acid, methacrylic acid, and anhydrides or esters derived from these, or mixtures thereof. Maleic anhydride is particularly preferred.

The coupling agents can be used as they are or pregrafted onto a polyolefin, for example polyethylene or copolymers of ethylene with an  $\alpha$ -olefin, by means of a radical reaction (see for example patent EP-530,940). The amount of coupling agent grafted is generally



between 0.05 and 5 parts by weight, preferably between 0.1 and 2 parts by weight, relative to 100 parts by weight of polyolefin. Polyolefins grafted with maleic anhydride are available as commercial products known, for example, under the brand names Fusabond® (Du Pont), Orevac® (Elf Atochem), Exxelor® (Exxon Chemical), Yparex® (DSM), etc.

Alternatively, the coupling agents of carboxylic or epoxide type mentioned above (for example maleic anhydride) or the silanes with ethylenic unsaturation (for example vinyltrimethoxysilane) can be added to the mixture in combination with a radical initiator so as to graft the compatibilizing agent directly onto the polymer matrix. An organic peroxide such as tert-butyl perbenzoate, dicumyl peroxide, benzoyl peroxide, di-tert-butyl peroxide can, for example, be used as initiator. This method is described, for example, in patent US-4,317,765, in Japanese patent application JP-62-58774 or alternatively in the abovementioned European patent applications Nos. 97121042.2 and 98118194.4.

The amount of coupling agent to be added to the mixture can vary mainly depending on the type of coupling agent used and on the amount of flame-retardant filler added, and is generally between 0.01 and 5%, preferably between 0.05 and 2%, by weight relative to the total weight of the base polymer mixture.

Other conventional components such as antioxidants, processing coadjuvants, lubricants, pigments, other fillers can be added to the compositions according to the present invention.

Conventional antioxidants which are suitable for this purpose are, for example: polymerized trimethyldihydroquinoline, 4,4'-thiobis (3-methyl-6-tert-butyl)phenol; pentaerythryl tetra-[3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate], 2,2'-thiodiethylene bis[3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate], or mixtures thereof.

Other fillers which may be used in the present invention include, for example, glass particles, glass fibres, calcined kaolin, talc, or mixtures thereof. Processing co-adjuvants usually added to the polymer base are, for example, calcium stearate, zinc stearate, stearic acid, paraffin wax, silicone rubbers, or mixtures thereof.

The flame-retardant compositions according to the present invention are preferably used in non-crosslinked form, in order to obtain a coating with thermoplastic properties which is thus recyclable.

The flame-retardant compositions according to the present invention can be prepared by mixing the polymer base, the flame-retardant filler, the dehydrating agent and the other additives which may be present according to techniques known in the art, for example using an internal mixer of the type containing tangential rotors (Banbury) or interlocking rotors, or in continuous mixers of the Ko-Kneader (Buss) type or of the co-rotating or counter-rotating twin-screw type.

Preferably, the dehydrating agent is introduced after a first phase of processing the composition during which, on account of the heating generated by the compounding process, the flame-retardant filler loses a certain amount of the moisture absorbed. In this way, premature depletion of the water-absorbing capacity of the dehydrating filler is avoided, this filler needing to be active mainly during the subsequent extrusion phase. The temperature of the composition in this first compounding phase is at least 100°C, preferably at least 150°C, and is carried out for a period of at least 5 minutes.

Alternatively, rather than adding the dehydrating agent during the phase of preparation of the flame-retardant composition, it can be added during the extrusion phase, for example via the extruder hopper.

In both cases, the dehydrating agent can be added to the flame-retardant composition in divided form (granules, powder), optionally coated with dispersing and protective agents, such as microwaxes, fatty acids. 5 Alternatively, in order to improve its dispersion in the polymer base, the dehydrating agent can be used predispersed in a polymer material (for example a semi-crystalline ethylene/propylene rubber).

During the extrusion phase, the flame-retardant 10 compositions thus obtained can be used to coat the conductor directly, or to make an outer sheath on the conductor which has been precoated with an insulating layer. When two layers are present, the extrusion can take place in two separate phases, the inner layer being 15 extruded on the conductor in a first passage and the outer layer being extruded on the inner layer in a second passage. Advantageously, the coating process can take place in a single passage, for example by means of the "tandem" technique, in which two separate extruders 20 arranged in series are used, or alternatively by co-extrusion with a single extrusion head.

The temperature at which the flame-retardant composition is extruded can vary within a wide range and is predetermined as a function of the extrusion rate to 25 be obtained. The extrusion rate in fact depends on the viscosity of the composition in the molten state and thus on its temperature. In turn, the viscosity depends mainly on the type of polymer base and on the type and amount of flame-retardant filler used. The minimum 30 extrusion temperature for the composition is generally not less than the plasticization temperature of the polymer base, while the maximum extrusion temperature is predetermined so as to avoid degradation or decomposition of the polymer base and/or of the flame- 35 retardant filler. Thus, on the basis of the abovementioned criteria, in the case of flame-retardant compositions based on a mixture of polypropylene and

and mixtures thereof. Examples of such polymers are: polyethylene (PE), in particular linear low-density PE (LLDPE); polypropylene (PP); propylene/ethylene thermoplastic copolymers; ethylene/propylene rubbers (EPR) or ethylene/propylene/diene rubbers (EPDM); natural rubbers; butyl rubbers; ethylene/vinyl acetate (EVA) copolymers; ethylene/methyl acrylate (EMA) copolymers; ethylene/ethyl acrylate (EEA) copolymers; ethylene/butyl acrylate (EBA) copolymers; ethylene/ $\alpha$ -olefin copolymers.

Alternatively, a self-extinguishing cable which can be made according to the present invention can consist of a conductor coated directly with the flame-retardant composition, without interposition of other insulating layers. In this way, the flame-retardant coating also functions as an electrical insulator. A thin polymer layer which functions as an anti-abrasive agent, optionally combined with a suitable pigment in order to give a coloration for identification purposes, can then be added externally.

#### Preparation of the flame-retardant compositions

The flame-retardant compositions were prepared in a closed Banbury mixer (mixing chamber volume: 1200 cm<sup>3</sup>) filled to a volumetric level of 95 %. The mixing was carried out in two phases. In the first phase, the components of the compound, with the exception of the dehydrating agent, were mixed together until a temperature of about 200°C was reached, so as to ensure good dispersion of the components and to reduce the amount of moisture present in the filler. The dehydrating agent was then added, while keeping the mixing temperature at about 200°C.

#### Mechanical properties

Self-extinguishing cables were produced by extruding the compositions prepared as described above on a wire of red copper (cross-section 2.5 mm<sup>2</sup>) in an extruder with a cylinder 120 mm in diameter and with a

CLAIMS

1. Process for producing a self-extinguishing cable, which comprises:
  - (a) preparing a flame-retardant composition comprising a polymer base, an inorganic flame-retardant filler and a dehydrating agent;
  - (b) extruding said flame-retardant composition on an electrical conductor, which is optionally precoated with an insulating layer, so as to obtain a flame-retardant coating;characterized in that preparing the flame-retardant composition comprises:  
mixing the polymer base with the inorganic flame-retardant filler at a predetermined temperature and for a predetermined time so as to reduce moisture contained in the flame-retardant filler, and then  
adding the dehydrating agent to the resulting mixture.
2. Process according to Claim 1, wherein the dehydrating agent is added during phase (a) of preparation of the flame-retardant composition.
3. Process according to Claim 1, wherein the dehydrating agent is added during phase (b) of extrusion of the flame-retardant composition.
4. Process according to any one of the preceding claims, wherein the polymer base is mixed with the inorganic flame-retardant filler at a temperature of at least 100°C for a time of at least 5 minutes.
5. Process according to any one of the preceding claims, wherein the dehydrating agent is added to the flame-retardant composition in divided form.
6. Process according to any one of the preceding claims, wherein the dehydrating agent is added to the flame-retardant composition after first being dispersed in a polymer material.

7. Process according to any one of the preceding claims, wherein the flame-retardant composition is extruded at a predetermined temperature which depends on the extrusion rate to be obtained.
8. Process according to Claim 7, wherein the flame-retardant composition is extruded at a temperature of between 160°C and 320°C.
9. Process according to Claim 8, wherein the flame-retardant composition is extruded at a temperature of between 200°C and 280°C.
10. Process according to any one of the preceding claims, wherein the dehydrating agent is chosen from: calcium oxide, calcium chloride, anhydrous alumina, zeolites, magnesium sulphate, magnesium oxide, barium oxide, or mixtures thereof.
11. Process according to Claim 10, wherein the dehydrating agent is chosen from: calcium oxide and zeolites, or mixtures thereof.
12. Process according to any one of the preceding claims, wherein the dehydrating agent is added to the flame-retardant composition in an amount of between 0.5 and 15 % by weight relative to the weight of the flame-retardant filler.
13. Process according to Claim 12, wherein the dehydrating agent is added to the flame-retardant composition in an amount of between 1 and 10 % by weight relative to the weight of the flame-retardant filler.
14. Process according to any one of the preceding claims, wherein the flame-retardant filler is chosen

from: hydroxides, hydrated oxides, salts or hydrated salts of metals.

15. Process according to Claim 14, wherein the flame-retardant filler is chosen from: magnesium hydroxide, alumina trihydrate, hydrated magnesium carbonate, magnesium carbonate, hydrated calcium and magnesium carbonate, calcium and magnesium carbonate, or mixtures thereof.

16. Process according to Claim 15, wherein the flame-retardant filler is magnesium hydroxide.

17. Process according to Claim 15, wherein the flame-retardant filler is magnesium hydroxide of natural origin.

18. Flame-retardant composition comprising:

(a) a copolymer of ethylene with at least one  $\alpha$ -olefin containing from 3 to 12 carbon atoms, and optionally with a diene, having a density of between 0.860 and 0.904 g/cm<sup>3</sup> and a composition distribution index greater than 45 %, said index being defined as the percentage by weight of the copolymer molecules having an  $\alpha$ -olefin content of up to 50 % of the total average molar content of  $\alpha$ -olefin;

(b) natural magnesium hydroxide;

(c) a dehydrating agent.

19. Composition according to Claim 18, wherein the dehydrating agent is chosen from: calcium oxide, calcium chloride, anhydrous alumina, zeolites, magnesium sulphate, magnesium oxide, barium oxide, or mixtures thereof.

20. Composition according to Claim 19, wherein the dehydrating agent is chosen from: calcium oxide and zeolites, or mixtures thereof.

21. Composition according to any one of Claims 18 to 20, wherein the dehydrating agent is present in an amount of between 0.5 and 15 % by weight relative to the weight of the flame-retardant filler.

22. Composition according to Claim 21, wherein the dehydrating agent is present in an amount of between 1 and 10 % by weight relative to the weight of the flame-retardant filler.

23. Composition according to any one of Claims 18 to 22, wherein the copolymer of ethylene with at least one alpha-olefin, and optionally with a diene, is obtained by a single-site catalyst.

24. Composition according to Claim 23, wherein the copolymer of ethylene with at least one alpha-olefin, and optionally with a diene, is obtained by a metallocene catalyst.

25. Composition according to Claim 23, wherein the copolymer of ethylene with at least one alpha-olefin, and optionally with a diene, is obtained by a constrained geometry catalyst.

26. Composition according to any one of Claims 18 to 25, further comprising a crystalline propylene homopolymer or copolymer.

27. Composition according to any one of Claims 18 to 25, further comprising an ethylene homopolymer or copolymer having a density of between 0.905 and 0.970 g/cm<sup>3</sup>.

28. Composition according to any one of Claims 18 to 27, further comprising a coupling agent to enhance compatibility between the flame-retardant filler and the polymer base.



29. Composition according to Claim 28, wherein the coupling agent is chosen from: saturated silane compounds; silane compounds containing at least one ethylenic unsaturation; epoxides containing an ethylenic unsaturation; monocarboxylic acids or dicarboxylic acids and derivatives thereof having at least one ethylenic unsaturation.

30. Process for producing a flame-retardant composition, which comprises:  
mixing a polymer base with an inorganic flame-retardant filler at a predetermined temperature and for a predetermined time so as to reduce moisture contained in the flame-retardant filler, and then  
adding a dehydrating agent to the resulting mixture.

31. Process according to Claim 30, wherein the polymer base is mixed with the inorganic flame-retardant filler at a temperature of at least 100°C for a time of at least 5 minutes.

32. Cable comprising a conductor and at least one flame-retardant coating layer, characterized in that said at least one flame-retardant coating layer comprises a composition according to any one of Claims from 18 to 29.

## PCT

## INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference <b>PC792</b>	<b>FOR FURTHER ACTION</b> see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
International application No. <b>PCT/EP 99/ 10131</b>	International filing date (day/month/year) <b>20/12/1999</b>	(Earliest) Priority Date (day/month/year) <b>24/12/1998</b>
Applicant <b>PIRELLI CAVI SISTEMI S.p.a et al</b>		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 2 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

**1. Basis of the report**

- a. With regard to the language, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

☐ the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

- b. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the international search was carried out on the basis of the sequence listing:

☐ contained in the international application in written form.

☐ filed together with the international application in computer readable form.

☐ furnished subsequently to this Authority in written form.

☐ furnished subsequently to this Authority in computer readable form.

☐ the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

☐ the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

2. ☐ Certain claims were found unsearchable (See Box I).

3. ☐ Unity of invention is lacking (see Box II).

4. With regard to the title,

☒ the text is approved as submitted by the applicant.

☐ the text has been established by this Authority to read as follows:

5. With regard to the abstract,

☒ the text is approved as submitted by the applicant.

☐ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the drawings to be published with the abstract is Figure No.

☐ as suggested by the applicant.

☐ because the applicant failed to suggest a figure.

☐ because this figure better characterizes the invention.

☐ None of the figures.

## INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 99/10131

**A. CLASSIFICATION OF SUBJECT MATTER**  
 IPC 7 H01B7/295 C09K21/02

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 H01B C09K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	W0 83 04041 A (NORSK KABELFABRIK AS) 24 November 1983 (1983-11-24)  page 3, line 7-17 page 12, line 5-9; claims 1-34	1-3, 5-15, 18-24
X	EP 0 718 388 A (BALLE ERIKA ;OTTO EUGEN (DE); OTTO UTA DR (DE)) 26 June 1996 (1996-06-26) page 5, line 49-59 page 11, line 49-51; claims 1,13	1-3,6,7, 12,14,18

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

## \* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

6 April 2000

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13/04/2000

Name and mailing address of the ISA

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# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/EP 99/10131

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